

**Development of epoxy-geopolymer hybrid composites as synthetic
ceramic**

by

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15225

Dissertation submitted in partial fulfillment of

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Universiti Teknologi PETRONAS

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Chemical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfillment of the requirement for the
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Approved by,

(AP DR. ZAKARIA BIN MAN)

Universiti Teknologi PETRONAS

TRONOH, PERAK

MAY 2014

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MUHAMMAD HAFIZ BIN BAHARUDIN

ABSTRACT

Novel cross-linked geopolymer-epoxy hybrid composites as synthetic ceramics is developed in this project. Geopolymer fly-ash is used as basis to be mixed with epoxy, an inorganic polymer at different ratios to produce an enhanced sample with lighter, strong resistance against temperature, acidity, low water absorption as well as high flexural strength. The project is carried out in order to suggest a solution of the expensive ceramics, as well as the high cost of manufacturing. Fly ash geopolymer is very cheap as they are the by-products of combustion which is not of any valuable use currently. Besides that the starting material of the sample is also very environmental friendly as it is biodegradable as well as inexpensive. Before starting on the preparation, multiple ratios of the geopolymer and epoxy will be studied to identify the best ratio. After the analysis, it is shown that 10% epoxy is the best formulation due to its least amount of water absorption, good surface as well as high density of material. The addition of epoxy has improved the properties of geopolymer.

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INTRODUCTION

1.1 Background of Study

Globally, polymer industry has started to emerge after the development of petroleum exploration. The plastics that are durable, flexible, light weight, strong mechanical and chemical properties, high resistance of heat, vibration, noise, corrosion, water and moisture along with the large variation of finishes and textures makes them able to be utilized in both hidden and visible applications (Zuhua Zhang, 2009).

Geopolymers is the term that refers to a class of earthly materials that is aluminium silicate inorganic polymers. Binders of geopolymer and cements are usually formed through the reaction of aluminium and silicon sources containing AlO_4 and SiO_4 tetrahedral units with high alkaline conditions (Provis & Deventer, 2009).

In the formation of geopolymers, metakaolin is an example of a common aluminosilicate starting material. It is easy to be handled as it has predictable physical properties. Other than that, fly ash which is a by-product of coal combustion can also be used as a material in the formation of geopolymer. A typical geopolymer have the following general formula $\text{Mn} [-\{\text{Si} - \text{O}_2\}_z - \{\text{Al} - \text{O}_2\}_n]$. The synthesis of geopolymers are through the combination of the aluminosilicate powder and alkaline solution (Provis & Deventer, 2009).

The initial applications of these geopolymers are coatings as it has a high thermal-resistance and also monolithic refractory. Its primary applications have been shifted, as researches increase in numbers and depth, to applications in the construction industry as it is possible to produce a high-performance geopolymers through the alkaline activation. Nevertheless, geopolymers are brittle and have a low flexural strength thus making it a less favourable material for application of structural material. Therefore, another organic polymer is incorporated into the geopolymer-based material to increase its toughness and strength (Ferone et al, 2012). In this paper, epoxy resins, which is the chemical compound diglycidyl ether bisphenol A, is incorporated with the inorganic geopolymer, fly ash.

1.2 Problem Statement

Current ceramics are strong and have a high temperature resistance. However the production of ceramics requires a high temperature of up to 1000°C. This process can be very costly as to achieve this temperature. Besides that the formation of ceramics may take a long time. Hence there is a need to find a new solution where this material can be produced at moderate-cost, easier handling, lower temperature and less time needed for production. This project is needed as a salvation to the problems above that is to produce a more beneficial starting material with moderate properties.

1.3 Objectives

The aim and objectives of this research experiment is as follows:

- i. Preparation and characterization of geopolymer
- ii. Geopolymer-epoxy formulation and preparation of sample
Determination of the most effective ratio of fly ash-epoxy
- iii. Characterization of the epoxy-geopolymer hybrid through water uptake test, density test and microstructure analysis

1.4 Scope of Study

To achieve the following objectives, the scopes of study which are related to the research experiment is as below:

- i. Preliminary Literature Review
Theoretical and analytical studies on recent previous researches relating to epoxy, polymer, geopolymer as well as epoxy-geopolymer hybrid composites.
- ii. Characterisation and analysis test
Water absorption test, microstructure analysis and density test.

LITERATURE REVIEW

2.1 Construction Industry

Ceramic products such as mortar and plaster, bricks, mosaic and decorative tile are a common and major part of the construction industry since the time of the Greek civilization (Richerson, 2005). These tiles are very durable and hygienic construction that adds attractiveness to the building, typically the bathrooms which are covered with these tiles.

Besides that, certain conditions favour the use of ceramics over concrete for example, structures exposed to high temperature fluctuations. The structure has to be able to withstand very high and low temperature from launch of space shuttle or structures built in the ocean.

Mechanism	Applications
Simple Substitution	Road Pavement and Bridge Decks Fire Protection for Steel Pipe and Pipe Linings
Demanding Environments	Airfield Pavements Walls for Engine Testing Facilities Space Structures Vessels, Reactors and Conduits for High Purity and Hazardous Substances
Fundamental Changes in Construction	Self-monitoring Containment or Reactor Chloride Trapping Bridge Deck

Table 1: Proposed applications for advanced ceramics in construction using three mechanisms of introduction (“The Potential for Advanced Ceramic Materials in Construction,” 1987)

The mechanism as shown above in how the ceramics are possibly introduced into the construction industry is by simple substitution of material, applications in structure operating under demanding environment and possible fundamental changes in construction. In time, the use of ceramics in construction industry will be introduced in a wider range of construction applications particularly in applications that require low maintenance and high performance.

However, the market for ceramics in the construction industry is seen from the current construction problems. Construction problems nowadays include the infrastructural decay in the industry, which is seen from the limited material life for many concrete structures due to exposure to marine environment (Vivas et al, 2007). The high cost of restoration opens up opportunity for a market of materials that is more long-lasting and requires least maintenance. There is also another challenge that is the containment of hazardous waste. High performance materials with green or better controlled microstructures are to be developed in order to use this kind of material (Steveson & Crentsil, 2005).

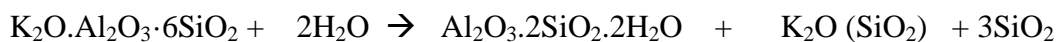
Alternatives to organic matrices for composites are being investigated particularly on geopolymer. Polymineral resins are the basis of the matrix, which are alumino-silicate binders. Although these binders are manufactured at low temperature, it can withstand ultra-high temperatures up to 1200°C. Besides that, this resin significantly improves the compressive strength and toughness as well as a lower shrinkage (Colangelo et al, 2013). These properties make them even durable and long-lasting even in marine environment. In addition to the purpose of manufacturing tiles, sinks, kitchenwares and other ceramic based products, the incorporation will make the material lightweight and contributes to the ease of handling.

In this paper, an alternative material of producing the kitchenware, sinks and tiles is to be produced, that is the geopolymer fly ash, which is incorporated with epoxy resins diglycidyl ether of bisphenol a (DGEBA), is proposed to be of high availability, low cost, lightweight as well as high thermal resistivity which could perform better and in addition, outweighing the cost.

2.2 Ceramic

Ceramics have been used long ago since ancient times. Man-made ceramics have been discovered by archaeologists that date back to at least 24,000 BC (Vandiver et al, 1989). In earlier times, ceramics were made of earthly materials such as clay and were burnt in domes. Intuitiveness progressively started with burning the materials at higher temperature increasing the hardness of ceramic. The desire for attaining harder substances has led to the invention of improved firing techniques. The ceramics are also biodegradable as they are made 100% from earthen materials (Cameron et al, 1977). They don't decay as result of natural impacts and are green materials. These properties surpass its brittleness as it is a strong-fragile part of human life.

Ceramics are defined as inorganic materials that are usually produced from clays and other earthen materials or chemically processed powders. However ceramics can also be compounds formed between metallic and non-metallic materials such as aluminium oxide and are crystalline in nature (Rosso, 2006). They are lightweight, high fire-resistant, and strong.



Feldspar

a clay mineral in solution in the clay

The clay is formed due to the deposition elsewhere which is carried by rivers. The particles are grounded finer when transported by water and through the action of other rocks. Separation of clay by size and settlement results in the different properties of clay due to the applied conditions during formation (Breuer, 2012).

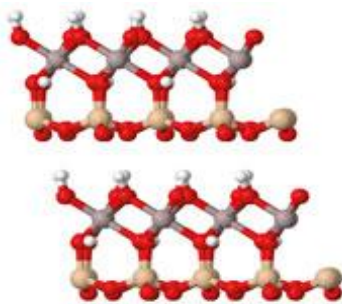


Figure 1: Part structure of kaolinite: Si - buff, Al - grey, O - red, H – white (The Chemistry of Pottery, 2012).

In comparison between the traditional ceramics and advanced ceramics, the method of manufacturing has been improved significantly with new technology development as well as a better understanding of advanced ceramic chemistry. In this century, a major development was reported by Garvie et al, whereby partially stabilised zirconia and toughening of the ceramic through phase transformation is developed (Taylor, 2001).

In these modern times, ceramics have become more advantageous over metals due to their ability to withstand elevated temperatures. It is one of the essential properties that replace metal in a gas turbine engine (Richerson, 2005). Ceramic rotor has less inertia thus causing the rotor to accelerate more rapidly. They are also anti-corrosive and high resistant to oxidation. The hardest substances known are ceramics, such as diamond and silicon carbide. Besides that ceramics can also be used in bearings without the need of lubricant, as to its low coefficient of friction, wear-resistant and high compressive strength.

Ceramics have become such an essential part of our life in the modern world. Many industries are using ceramics as its key components in the materials. For example, in the making of a space shuttle, ceramics are used due to its light weight thermal barrier properties as the edges of the wings and for the nose cap. Ceramics has high tension insulation, which makes it is plausible to carry electricity to houses and businesses. Not to mention in the medical field, where ceramics are being used explicitly as prosthetics, bone repair, dental as well as treatment (Richerson, 2005).

The project's application is focused on the interior of building such as tiles, sinks, table tops and kitchen wares. Therefore the properties of the material varies than the other applications, which is a material that is more light weight, reduced capital cost, easier handling as well as lower temperature of manufacturing.

2.3 Geopolymer

The condition of forming geopolymers, which are synthetic inorganic aluminosilicate materials, is by reacting aluminosilicate with silicate solution in strong alkali such as sodium hydroxide. Polymeric precursors are produced by the repetition of two tetrahedral units of silica oxide and alumina oxide, by sharing all oxygen atoms ($-\text{SiO}_4-\text{AlO}_4-$) (Roviello et al, 2013).

Geopolymers have a high potential of becoming the substitute for ceramics as it is inexpensive, waste products are being used such as fly ash or blast furnace slags. Besides that, these materials are environmental friendly as industrial solid wastes are made full use (Roviello et al, 2013). Geopolymers also have a higher resistant of fire and acid and less shrinkage. A shorter period of time is required to develop a high strength of polymer.

The properties and fields of application is determined by the atomic ratio of silica to aluminium (Si-O-Al-O) structure which are the poly (sialate), the basis of the matrix composite (Duxson, 2005). In order to serve as an alternative of ceramic, the ratio of 1:1 can be used, to get a rigid three-dimensional network.

In the synthesis of geopolymer, an alkaline activating solution was prepared. Solid sodium hydroxide is dissolved into the sodium silicate solution. Equilibrium is then allowed to be reached and cooled. The solution's composition can be expressed as $\text{Na}_2\text{O} \ 1.4\text{SiO}_2 \ 10.5\text{H}_2\text{O}$. Fly ash is then added to the solution with a ratio of solid to liquid 1:1.4 respectively by weight and mixed manually for 15 minutes. The geopolymer's composition can be expressed as $\text{Al}_2\text{O}_3 \ 3.5\text{SiO}_2 \ 1.0\text{Na}_2\text{O} \ 10.4\text{H}_2\text{O}$, with assumption of 100% geopolymerization (Roviello et al, 2013). Although geopolymer needs a shorter time to reach a high strength, its mechanical properties are similar to that of other organic composites such as ceramic.

The starting material of fly ash, which is waste from combustion of coal, is used. Therefore the natural resources have a lower depletion due to artificial aggregates being waste products (Roviello et al, 2013). This makes the material of geopolymer an environmental friendly aside from being low cost and high availability.

Nevertheless, geopolymers have a low flexural strength and brittle makes it a less favourable material for application of structural material (Colangelo, 2013). Therefore, another organic polymer is incorporated into the geopolymer-based material to increase its toughness and strength. In this paper, epoxy resins, which is the chemical compound diglycidyl ether bisphenol A, is incorporated with the inorganic geopolymer, fly ash.

2.4 Epoxy

Epoxy resin is widely used in electronic packaging industries as it is easy to be processed and low cost. Researches also widely use epoxy resins due to their high heat, moisture and chemical resistance (Kang et al, 2001). Although it is widely used, the epoxy resins are not suitable for the board dimensional control and the high coefficient of thermal expansion mismatch between the substrate and silicon chip which leads to solder joint failures and due to the distortion during thermal cycling.

In the early 1960's, ceramic materials have been assembled into the electronic packaging industry due to its relatively low coefficient of thermal expansion which is near to the silicon chips. However the main factors which are the cost and ease of processing. Hence, the low coefficient of thermal expansion ceramic fillers is added into the polymeric materials to reduce the coefficient of thermal expansion and increase the elastic modulus of the resins (Kang et al, 2001). In order to achieve an ultra-high density wiring and to assemble area array flip-chips with minimal stress on solder joint, a high elastic modulus is required. The coefficient of thermal expansion of the polymer composites is reduced by adding a higher amount of fillers content.

There is however a problem during the processing if too much filler is added. This is due to the high viscosity of commercial epoxy resins based on bisphenol A. About 40 volume percentage is the maximum loading percentage of silica filler in bisphenol A epoxy resin (Teh et al, 2007).

Mortar and pestle is the mechanical technique used in order to produce the material below. A volume percentage of 80, 85, 90 and 95 which is the maximum mineral silica loading in bisphenol A epoxy resin can be reached by using this method. During mixing, mechanical friction occurs when the resins and fillers are thoroughly mixed with the resin covering the ceramic filler particle. The viscosity of

the sample will drop when the samples undergo curing (Cicala et al, 2005). The gap between the filler is filled up by the resins by capillary action.

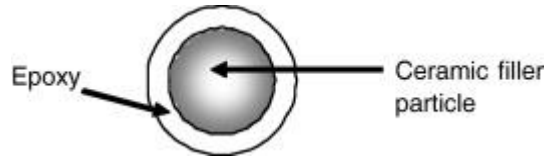


Figure 2: Polymer coated ceramic filler particle

Epoxy resin is also the most widely used material for composites of aircraft structures. Due to its low shrinkage, stiffness, good adhesion to carbon, fibre, glass, etc. Unfortunately, the factor contributing to its heat resistance and high stiffness is the source of its drawback that is the lack of toughness (Park et al, 2004)

2.5 Epoxy-Geopolymer Hybrid Composites

In order to overcome the problems above, which are the high cost and temperature of manufacture, brittleness and durability, a hybrid of the above components are developed, which is the organic-inorganic matrix composites (Menna et al, 2013). Several kinds of organic polymers, each incorporated into geopolymers, have been investigated such as polyvinyl acetate, polyvinyl alcohol, polypropylene and water soluble organic polymers (Roviello et al, 2013). It was reported by Roviello that the hybrid composites of the two is different due to its compatibility with each other chemically.

The good compatibility between geopolymer and epoxy resins is achieved by promoting the formation of the highest number of hydroxyl tails in the epoxy ring opening reaction. This causes the organic epoxy phase to become temporarily hydrophilic and improves its chemical compatibility and bondage to the aqueous inorganic geopolymer phase (Roviello et al, 2013).

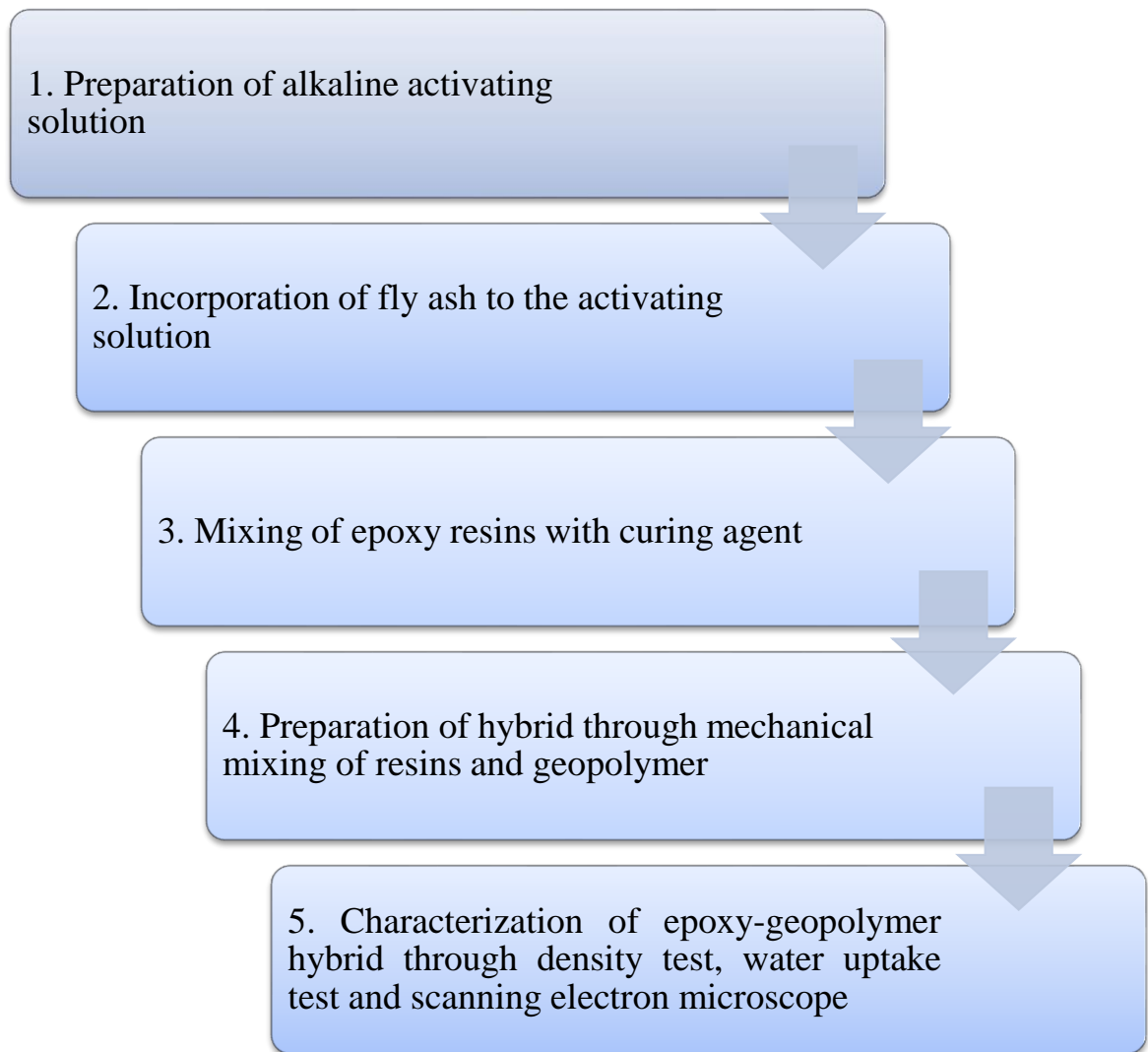
In comparison to the traditional geopolymer, these materials of organic resins and inorganic matrix composites are directed towards green chemistry as solvents are avoided completely. From the research of Ferone, it states that it is more environmental friendly as it has a longer service life due to its enhanced durability. As for the starting material, wastes from varieties of activities such as demolition, construction, mining, combustion and metallurgical are being used which leads to a reduction towards the environmental impact (Ferone et al, 2012).

Ferone have concluded that the new materials, being geopolymer-epoxy hybrid, present good technological properties that is increased toughness and compressive strength, which is 3.5 times the strength of geopolymer. Besides that, it was reported that the hybrid composites of geopolymer-epoxy show an enhanced thermal stability as well as an increased in fire performance capability (Hussain et al, 2004).

Further investigations are being studied in this paper as to determine the ratio of fly ash to epoxy that is most suitable in the application of house interiors. These applications require the properties of ease of manufacturing, low temperature of manufacturing, high durability, ease of handling, light weight, low shrinkage as well as low capital cost. These properties are characterized through density test, water absorption test and microstructure analysis.

METHODOLOGY

3.1 Research Methodology



3.2 Basic Experimental Procedures

3.2.1 Determination of Geopolymer-Epoxy Ratios

The research target is to improve the performance of fly ash based geopolymer. Hence, the amount of epoxy added is varied accordingly for analysis of each sample. The mixture of geopolymer consists of fly ash and the alkaline activating solution. The ratio of the mixture is 3:1. As for the mixture of epoxy, it consists of epoxy and amine with a ratio of 2:1. The mass of this mixture is calculated as below,

% Epoxy	Mass of fly ash (g)	Mass of NaOH (g)	Mass of Epoxy (g)	Mass of Amine (g)
0.00	100	33.3	0.0	0.0
5.00	100	33.3	4.7	2.3
10.00	100	33.3	9.9	4.9
20.00	100	33.3	22.2	11.1

Table 2: Mass of Epoxy needed for different % epoxy sample

3.2.2 Geopolymer-Epoxy Sample Preparation

The project research will be carried out by experimental procedures. This project has three major steps. Firstly, the preparation of the highly caustic compound, that is alkaline activating solution.

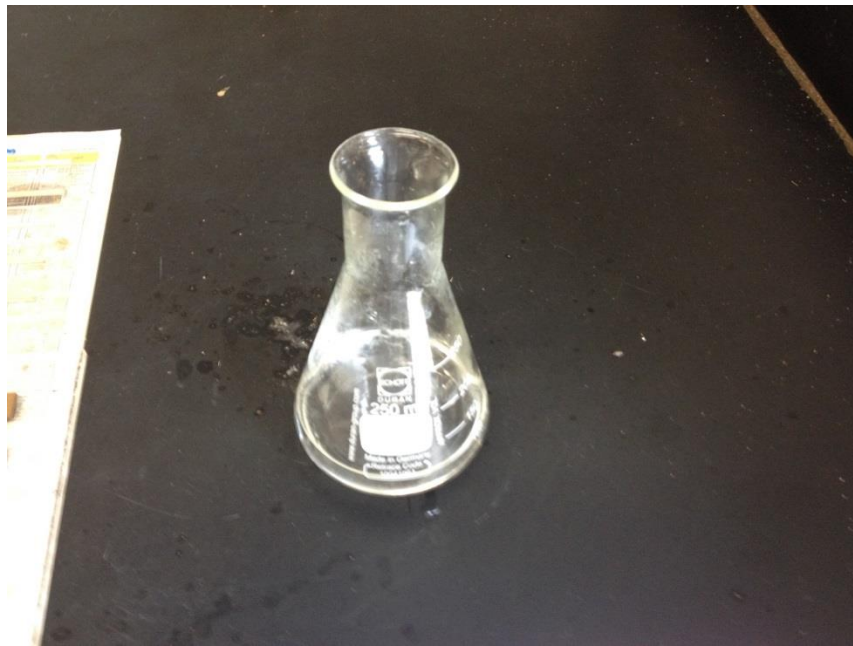


Figure 3: Preparation of alkaline activating solution

The solid sodium hydroxide is dissolved into the sodium silicate solution with a ratio of 85:15 respectively. The solution is then left to reach equilibrium and cooled for 24 hours. The solution's composition can be expressed as $\text{Na}_2\text{O} \ 1.4\text{SiO}_2 \ 10.5\text{H}_2\text{O}$.

After that, fly ash is incorporated into the activating solution with 3:1 weight ratio of solid to liquid and mixed manually for 15 minutes forming a geopolymer with a composition of $\text{Al}_2\text{O}_3 \ 3.5\text{SiO}_2 \ 1.0\text{Na}_2\text{O} \ 10.4\text{H}_2\text{O}$. This is with the assumption of 100% geopolymerization occurred.

Next, the curing agent and liquid epoxy resin are mixed in the second container for 60 minutes at room temperature, with a ratio of 2:1, epoxy to amine (curing agent).



Figure 4: The addition of curing agent to epoxy

The percentage of each component prepared is geopolymer varied accordingly from 0% epoxy to 20% epoxy. Finally, the two containers are blended together and mechanical mixing is quickly incorporated for 10 minutes and left to cure at 60°C for 1 hour and then left to cure at room temperature.

3.2.3 Analytical Processes

i. Water Absorption Test

After the sample has been prepared, the sample is characterized and analysed firstly through water absorption test. Currently, there are no standard to the exact procedure of measuring water uptake for geopolymer as ceramics. The test is to be carried out by weighing the initial weight of sample, followed by the immersion of sample in water for stipulated time at ambient temperature. Then the sample is dried and weighed again. These processes are to be repeated for several times. The equation below shows the formula which will be used to calculate the water absorption percentage by sample.

$$M_t = \frac{m_2 - m_1}{m_1} \times 100\%$$

Where;

M_t = Percentage of water absorption of sample

m_1 = Initial mass of sample in gram

m_2 = Final mass of sample in gram

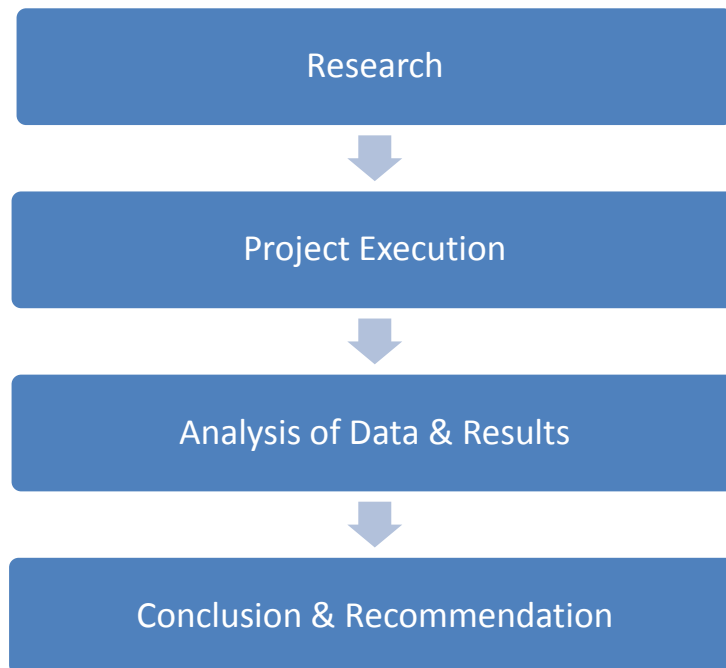
ii. Scanning Electron Microscope (SEM)

As for the microstructural analysis, it is done to investigate the morphology of the geopolymer-epoxy hybrid composite on the interfacial zone between them, as well as to investigate on the formation of aggregate. The formation of aggregate destabilises the colloidal system. The images will give a better visual on the properties of the materials.

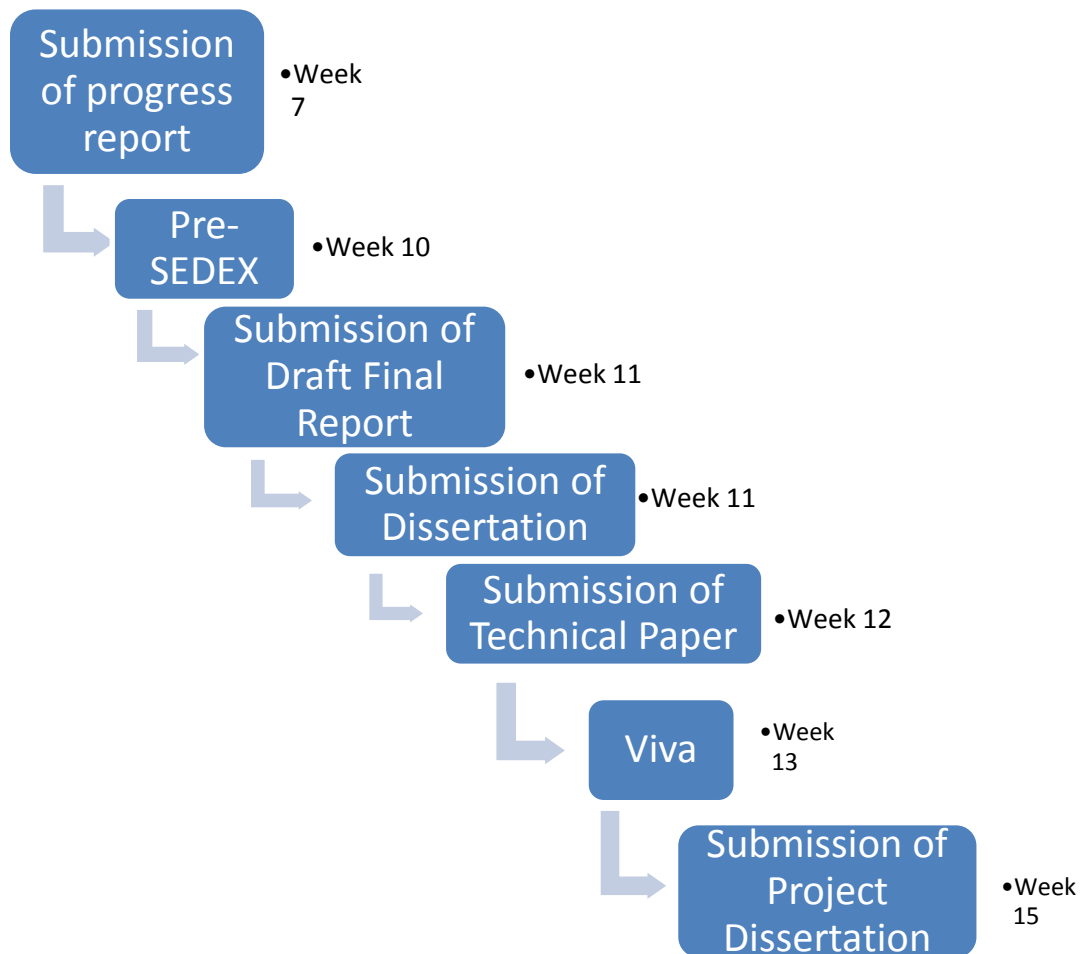
iii. **Density Test**

The density test is done by using the Ultrapycnometer 1000 Version 2.2. The equipment is used to determine the nitrogen and helium-based coal densities. The density measured is an average of the last 3 runs' value with standard deviation of less than 0.005. The value is reached through computations according to Boyle's law and Archimedes' principle of fluid displacement to determine the volume of sample. The gas which displaces fluid can penetrate very fine pores. Hence before the density the taken, the sample needs to be purged for 1 min.

3.3 Project Activities



3.3 Key Milestones



3.4 GANTT CHART

Timelines for FYP 2

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project Work Continues															
2	Submission of Progress Report															
3	Project Work Continues															
4	Pre-SEDEX															
5	Submission of Draft Final Report															
6	Submission of Dissertation (soft bound)															
7	Submission of Technical Paper															
8	Viva															
9	Submission of Project Dissertation (Hard Bound)															

RESULTS AND DISCUSSION

Epoxy is incorporated into fly ash at different percentages in this step. Multiple trials of preparation are carried out with the results of the following below.

4.1 Result of Sample Preparation

The sample is prepared with variation of epoxy that is 0%, 5%, 10% and 20%. The incorporation of epoxy into the 3:1 ratio of geopolymer fly ash to 8M of NaOH is used. The curing time is 24 hours at room temperature.

The geopolymer fly ash alone with no addition of epoxy is too brittle as they broke into smaller pieces, which shows that fly ash is not suitable in the replacing ceramics.

As for the geopolymer with 5% and 10% epoxy, the samples are in one piece. This proves the flexibility and strength is better in both of them.

Geopolymer with 20% epoxy is observed to be in excess of epoxy due to its glue-like appearance on the surface. The sample is very rough as more epoxy added, the sample becomes more viscous.

4.2 Result of FESEM Analysis

In this trial, the sample is prepared in the same way as before. The sample is to observe the surface roughness of the samples in a clearer picture.

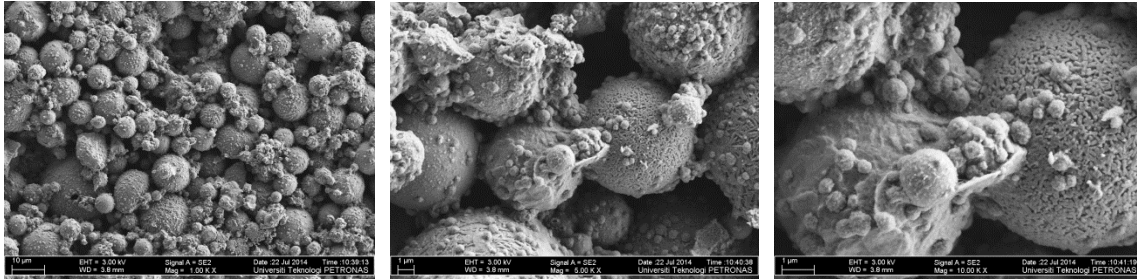


Figure 5 (a), (b) and (c): 1000x, 5000x and 10,000x magnification of 0% epoxy sample

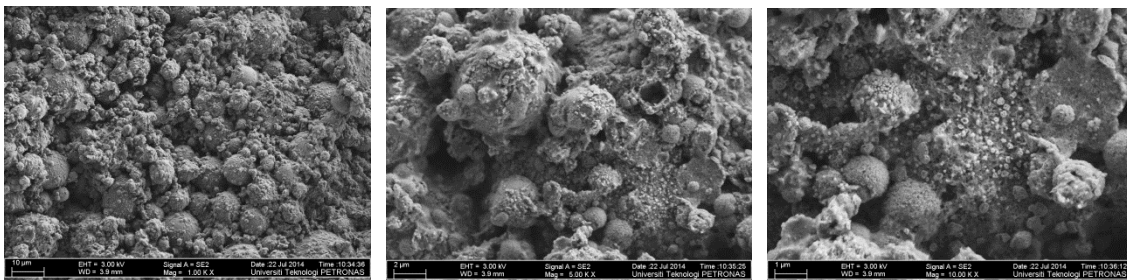


Figure 6 (a), (b) and (c): 1000x, 5000x and 10,000x magnification of 10% epoxy sample

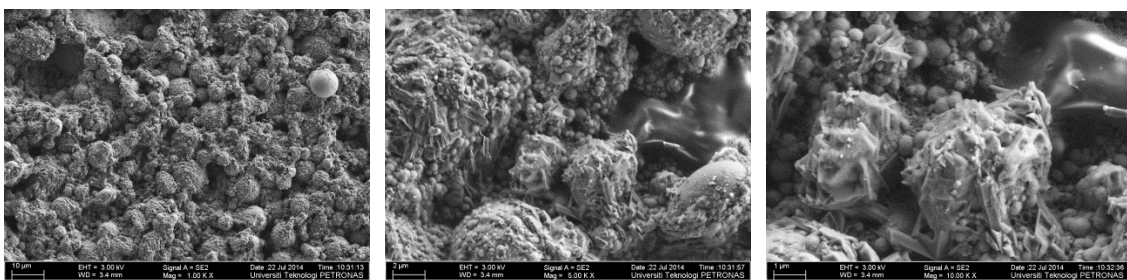


Figure 7 (a), (b) and (c): 1000x, 5000x and 10,000x magnification of 20% epoxy sample

As observed from the figures above, the surface roughness increases with increasing epoxy in the sample. It can also be seen that epoxy is not mixed well with each other. This is due to excess epoxy being added into the geopolymer.

4.3 Result of Water Absorption Test

The water absorption test is to measure how much water penetrates into the sample and being absorbed by the sample. It is crucial that the sample absorbs the least amount of water as to minimize deterioration of the properties as to replace ceramics. Below is the result for the test.

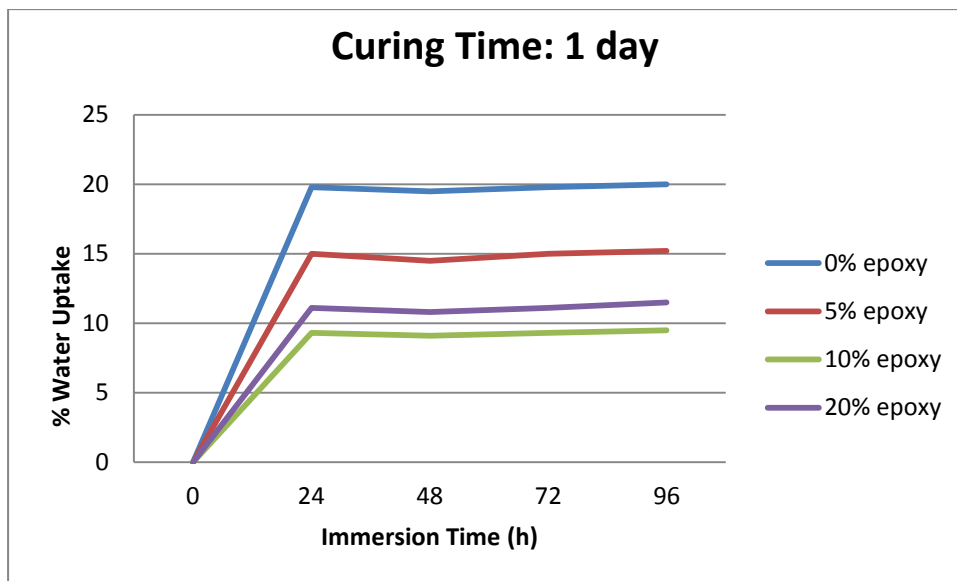


Figure 8: % water uptake for variation of % epoxy cured for 24 hours

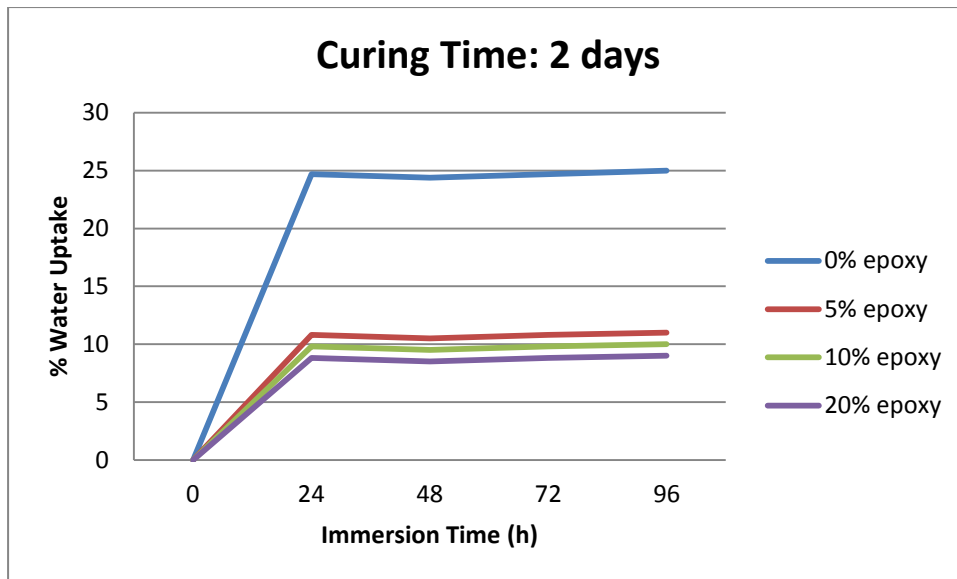


Figure 9: % water uptake for variations of % epoxy cured for 2 days

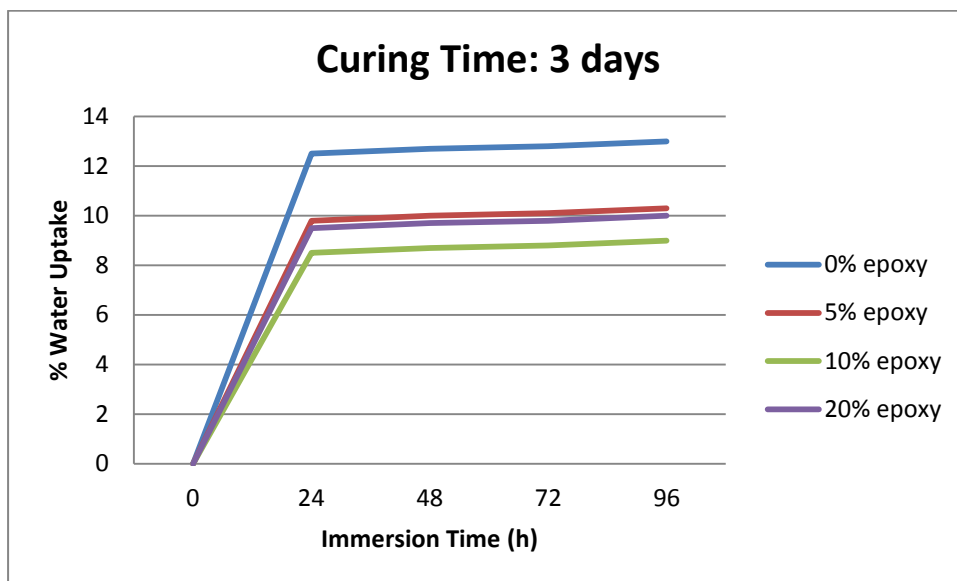


Figure 10: % water uptake for variations of % epoxy cured for 3 days

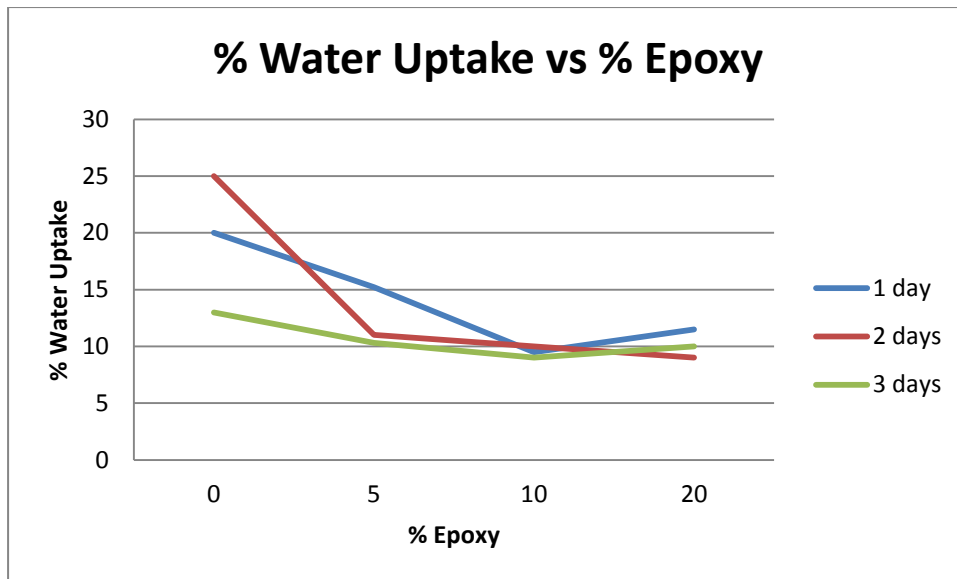


Figure 11: % water uptake against % epoxy for different curing time

Figure 11 shows the total result obtained from samples cured from different times (1, 2 and 3 days). There is no similar pattern to differentiate which curing time is better. However, it can be concluded that the least water absorbed is the sample with 10% epoxy cured at 3 days. This shows that a small amount of epoxy (10%) is required in enhancing the characteristic of the synthetic ceramic.

4.3 Density Test

The importance of this test is to identify which sample has the highest density. The result is used to estimate and identify which sample has the highest strength. The list of densities is as shown below.

Run	% Epoxy			
	0	5	10	20
1	3.4823	3.4631	3.5541	3.5765
2	3.5096	3.4629	3.5544	3.5769
3	3.5049	3.4635	3.5542	3.5773
4	3.5094	3.463	3.5549	3.5775
5	3.5149	3.4641	3.5551	3.5775
6	3.5041	3.4636	3.557	3.5773
Average	3.5042	3.4634	3.5550	3.5772

Table 3: Density for sample of different % epoxy

From the table above, it can be seen that sample with 20% epoxy have the highest density, followed with the 10% epoxy. This shows that increasing epoxy in the sample increases the density and strength.

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The addition of epoxy into the geopolymer fly ash does increase and enhance its properties and characteristics by decreasing the brittleness, pores, water uptake as well as increasing strength. The research is currently up to conclude that 10% epoxy is the best formulation due to its properties. Although the 20% epoxy has a higher density, the mixtures do not blend well together as the epoxy is in excess. This can be seen from Figure 7 (c). Hence, it can be concluded that the best percentage for epoxy to be in geopolymer is 10%.

5.2 Recommendations for Future Work

The geopolymer has a very wide scope and a lot more variables and analysis can be done. For starters, the flexural strength test can be done to measure the strength of the samples in more detail. Besides that, geopolymer can also be bind with many other inorganic polymers. This can be researched further to identify the best sample for the replacement of ceramics.

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